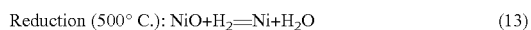
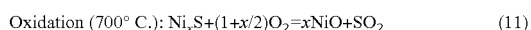


strated with the pure nickel sorbent. The regenerations were performed at 500° C. using 2% H₂ (for cycles 1 to 5) and 5% clean syngas (for cycles 6 to 8) as the reducing gas, and 10% air as the oxidation gas. Four redox treatments were carried out for each regeneration. Methane production with fresh Ni—Cu-SBA-16 was 0.16 mol %, vs. 0.7 mol % for the regenerated copper-free nickel adsorbent. No changes in concentration of CO, CO₂, and H₂ in the treated syngas were observed during sulfur removal. Pure Cu-SBA-16 was also evaluated for sulfur removal effectiveness. A high initial sulfur capacity was achieved (0.8 wt %), very similar to the Ni and Ni—Cu samples, but despite the confining pore structure of the SBA-16 the capacity reduced to less than 0.2% following regeneration, indicative of metal Cu sintering.

The Ni—Cu-SBA-16 sorbent (15 wt. % Ni, 1.6% Cu) was then tested for desulfurization of syngas simulated from a biomass gasifier. Test conditions: T=300° C.; biomass gas composition: 18% H₂, 12% CO, 10% CO₂, 50% H₂O, 4% He, 36 ppm H₂S; flow rate: 12,000 hr⁻¹ GHSV. Regeneration conditions: four “oxidation-reduction” treatments at 500° C. Oxidation in air at 14,000 hr⁻¹ GHSV. Reduction in clean dry syngas at 14,000 hr⁻¹ GHSV. Three minutes purge with Ar between oxidation and reduction treatment. The sulfur capacity with the biomass-based syngas is significantly higher than with the coal-based syngas, at 2.3 wt. % approximately a factor of 3 increase. A similar uptake capacity was also obtained with carbonyl sulfide was used as the sulfur gas. This is a very positive result, as adsorbents such as zinc oxide are less effective in removing COS than H₂S. The higher sulfur concentration in the feed likely contributes to these higher capacities (uptake capacity increases with H₂S partial pressure), but in addition the higher concentration of steam and lower concentration of CO may also contribute to the better performance. At this uptake capacity, sulfur removal cannot be explained simply by a surface adsorption mechanism, and bulk formation of nickel sulfide must be invoked which was clearly observed via XRD analysis of sulfur-loaded sorbent. The performance over multiple regeneration cycles is given in FIG. 8, showing that the oxidation-reduction regeneration procedure is equally effective with a bulk metal sulfide.

A simplified “oxidation-reduction” procedure was found to be as effective as the multi-cycle procedure is. This procedure requires only two steps: oxidation at 700° C. in air for 20 hours and reduction at 500° C. in a reducing gas for 4 hours, such as in diluted or no-diluted clean syngas stream. At 700° C., NiSO₄ and CuSO₄ are not stable; they decompose to metal oxides and SO₂. As a result, almost all the sulfur on the sorbent can be removed during this step. This new procedure can be easily integrated with the regeneration procedure of used ZnO-based sorbents, which also requires high temperature (~700° C.) oxidation of ZnS. Major reactions occur during this regeneration procedure include:



In the nickel and copper loading configurations described above a significant fraction of voids within the mesoporous structure of SBA-16 remain. This suggests that higher load-

ings of metal within the material are possible, providing a means to increase sulfur sorption capacity. At higher capacity, it is possible to operate the Ni—Cu-SBA-16 sorbent as a stand-alone device, without the need for an upstream zinc oxide bed.

In summary, by trapping Ni and Ni—Cu alloy nanoparticles in three dimensional mesostructured silica SBA-16, we have developed a class of metal-based adsorbents that can remove sulfur from gasifier-produced syngas from either coal or biomass to less than 50 ppb levels. A combination of sulfur chemisorption and (at higher uptakes) bulk sulfide formation appears to occur. A sequential oxidation-reduction treatment can effectively regenerate the sulfur-loaded adsorbents. This solid adsorbent-based approach can provide economic advantages compared with existing technologies based on ambient or lower temperature solvent-based cleanup systems. With coal-based syngas that may contain several thousand ppm of sulfur, these adsorbents could be used in combination with a higher capacity zinc oxide absorbent, providing the necessary sub-ppm polishing capability that cannot be provided by zinc oxide alone. With biomass-based syngas, which typically may contain 30-80 ppm sulfur gases, these sorbents could form the basis for a sub-ppm, stand-alone desulfurization system.

While various preferred embodiments of the invention are shown and described, it is to be distinctly understood that this invention is not limited thereto but may be variously embodied to practice within the scope of the following claims. From the foregoing description, it will be apparent that various changes may be made without departing from the spirit and scope of the invention as defined by the following claims.

What is claimed is:

1. A method of manufacturing regenerable sorbents to remove gas phase sulfur from warm fuel gases, comprising:
 - a. impregnating inorganic salts within a SBA-16 three dimensional mesoporous silica substrate; and
 - b. calcining and reducing the inorganic salts to obtain active metal-based sorbents within the substrate.
2. The method of claim 1 wherein the warm fuel gases include natural gas, syngas, H₂, CO, and hydrocarbon gases, mixtures of hydrocarbon gasses, and mixtures of hydrocarbon gases and inert gases, and wherein the warm fuel gasses have a temperature between 20 and 900 degrees C.
3. A method for desulfurizing a gas, comprising:
 - a. impregnating inorganic salts within a SBA-16 three dimensional mesoporous silica substrate;
 - b. calcining and reducing the inorganic salts to obtain active metal-based sorbents within the substrate; and
 - c. passing the gas over the substrate.
4. The method of claim 3 further comprising regenerating the active metal-based sorbents.
5. The method of claim 4 wherein the regenerating the active metal-based sorbents comprises subjecting the active metal-base sorbents to an oxidation-reduction process, utilizing oxidative gases that oxidize metal sulfides and reductive gas streams that reduce metal oxides.
6. The method of claim 5 wherein the oxidation-reduction process includes oxidation at approximately 700° C. in air for approximately 20 hours.